

FORM PTO-1390 (Modified) (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER <b>219861US0PCT</b>
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR <b>10/049623</b>	
INTERNATIONAL APPLICATION NO. <b>PCT/JP00/05510</b>	INTERNATIONAL FILING DATE <b>17 August 2000</b>		PRIORITY DATE CLAIMED <b>24 August 1999 (earliest)</b>	
TITLE OF INVENTION <b>WATER-CONTAINING POWDER COMPOSITION, PROCESS FOR PRODUCING THE SAME, AND COSMETIC PREPARATION CONTAINING THE POWDER COMPOSITION</b>				
APPLICANT(S) FOR DO/EO/US <b>TANAKA Yoichiro et al.</b>				
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:				
<ol style="list-style-type: none"> <li>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.</li> <li>4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</li> <li>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c) (2))             <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> </li> <li>6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).             <ol style="list-style-type: none"> <li>a. <input checked="" type="checkbox"/> is attached hereto.</li> <li>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</li> </ol> </li> <li>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))             <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input type="checkbox"/> have been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li>d. <input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li> <li>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).</li> <li>10. <input checked="" type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).</li> <li>11. <input type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409).</li> <li>12. <input checked="" type="checkbox"/> A copy of the International Search Report (PCT/ISA/210).</li> </ol>				
Items 13 to 20 below concern document(s) or information included:				
<ol style="list-style-type: none"> <li>13. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li> <li>14. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li> <li>15. <input type="checkbox"/> A <b>FIRST</b> preliminary amendment.</li> <li>16. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</li> <li>17. <input type="checkbox"/> A substitute specification.</li> <li>18. <input type="checkbox"/> A change of power of attorney and/or address letter.</li> <li>19. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</li> <li>20. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</li> <li>21. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</li> <li>22. <input type="checkbox"/> Certificate of Mailing by Express Mail</li> <li>23. <input checked="" type="checkbox"/> Other items or information:</li> </ol>				
<b>PCT/IB/304/Notice of Priority/PCT/IB/308</b> <b>Form PTO-1449/Amended Sheets (Pages 26 and 27)</b>				

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR <b>10/049623</b>	INTERNATIONAL APPLICATION NO. PCT/JP00/05510	ATTORNEY'S DOCKET NUMBER <b>219861US0PCT</b>																
24. The following fees are submitted:		<b>CALCULATIONS PTO USE ONLY</b>																
<b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :</b>																		
<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... <b>\$1040.00</b> <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... <b>\$890.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... <b>\$740.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... <b>\$710.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... <b>\$100.00</b>																		
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>		<b>\$890.00</b>																
Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)).		<input type="checkbox"/> 20 <input type="checkbox"/> 30 <b>\$0.00</b>																
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>CLAIMS</th> <th>NUMBER FILED</th> <th>NUMBER EXTRA</th> <th>RATE</th> </tr> </thead> <tbody> <tr> <td>Total claims</td> <td>9 - 20 =</td> <td>0</td> <td>x \$18.00</td> </tr> <tr> <td>Independent claims</td> <td>3 - 3 =</td> <td>0</td> <td>x \$84.00</td> </tr> <tr> <td colspan="2">Multiple Dependent Claims (check if applicable).</td> <td><input checked="" type="checkbox"/></td> <td><b>\$280.00</b></td> </tr> </tbody> </table>		CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	Total claims	9 - 20 =	0	x \$18.00	Independent claims	3 - 3 =	0	x \$84.00	Multiple Dependent Claims (check if applicable).		<input checked="" type="checkbox"/>	<b>\$280.00</b>	
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		<b>charged</b> <b>\$</b>																
a. <input checked="" type="checkbox"/> A check in the amount of <u><b>\$1,170.00</b></u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u><b>15-0030</b></u> A duplicate copy of this sheet is enclosed. d. <input checked="" type="checkbox"/> Fees are to be charged to a credit card. <b>WARNING:</b> Information on this form may become public. <b>Credit card information should not be included on this form.</b> Provide credit card information and authorization on PTO-2038.																		
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.																		
SEND ALL CORRESPONDENCE TO:																		
Surinder Sachar Registration No. 34,423  <b>22850</b>		 SIGNATURE <b>Norman F. Oblon</b> NAME <b>24,618</b> REGISTRATION NUMBER <b>Feb. 22 2002</b> DATE																

DESCRIPTION

WATER-CONTAINING POWDER COMPOSITION, PROCESS FOR PRODUCING  
THE SAME, AND COSMETIC PREPARATION CONTAINING THE POWDER

5 COMPOSITION

TECHNICAL FIELD

The present invention relates to a composition in which water is powderized and, more particularly, to a

10 water-containing powder composition that is superior in production stability and storage stability, and releases water when pressed with the fingers or the like during use. The water-containing powder composition is widely utilizable in cosmetics, foods, perfumes, agricultural chemicals, medicines, 15 and the like.

BACKGROUND ART

The inventors of the present invention previously developed a water-containing powder composition that releases 20 water when pressed with the fingers or the like during use, and filed a Patent application (Japanese Patent Application laid-open No. 1993-65212). This water-containing powder composition, prepared by a technology for powdering water and oil using powder treated with hydrophobic silicic acid 25 anhydride and a fluorine compound, experienced problems relating to stable production and storage stability, due to fluctuation in the degree of hydrophobicity of the silicic acid

anhydride.

Therefore, the inventors of the present invention investigated the technology of constantly producing the water-containing powder composition with the above properties 5 by using microcapsule technology.

The inclusion of water in hydrophobic particles using microcapsule technology has been disclosed in Japanese Patent Publication 1991-67737. In this method, water is frozen, crushed into particles, and then coated with hydrophobic 10 particles.

However, it is difficult to obtain a useful microcapsule using the above method due to the following problems: ① it is difficult to obtain a fine particle diameter when the frozen water is shattered; ② the water (ice) particles easily 15 flocculate and associate during the process of shattering frozen water and coating the particles; and ③ flocculation or association of the shattered water (ice) particles tends to occur when the surface of the particles is coated.

Therefore, development of a technology for manufacturing 20 a water-containing powder composition capable of releasing water when pressed with the fingers during use by improving conventional microcapsule technology without the above manufacturing problems has been desired.

## 25 DISCLOSURE OF THE INVENTION

As a result of extensive studies to solve the above problems, the inventors of the present invention have

discovered that a water-containing powder with a fine particle diameter can be obtained without the problems of flocculation and association of particles by causing an aqueous phase ingredient to gel with a water-soluble gellant, forming the 5 gel into particles each serving as a core by high speed shearing, freeze-shattering or the like, and then coating each core with hydrophobic particles. The inventors have further discovered that the obtained water-containing powder displays excellent production stability and storage stability, and maintains the 10 characteristics of releasing water when pressed with the fingers or the like during use. These findings have led to the completion of the present invention.

Specifically, the present invention provides a water-containing powder composition comprising aqueous gel 15 cores coated with hydrophobic particles.

The present invention further provides a process for manufacturing a water-containing powder composition comprising causing an aqueous phase ingredient to gel with a water-soluble gellant, forming the gel into particles each 20 serving as a core by means of high speed shearing or freeze-shattering, and coating the cores with hydrophobic particles.

Furthermore, the present invention provides a cosmetic composition comprising any one of the above mentioned 25 water-containing powder compositions and a method of applying the cosmetic compositions to the skin.

BEST MODE FOR CARRYING OUT THE INVENTION

The water-containing powder composition of the present invention has a structure wherein hydrophobic particles are coated on the exterior surface of aqueous gel cores. This water-containing powder composition is manufactured by causing an aqueous phase ingredient to gel with a water-soluble gellant, forming the gel into particles, each serving as a core, by high speed shearing, freeze-shattering or the like, and then coating each core with hydrophobic particles.

10        The amount of water used in the aqueous gel of the water-containing powder composition of the present invention, is preferably 30-99.7 weight% (hereinafter indicated by %) and more preferably 40-98%. The water content in this range can provide an excellent fresh feel of water.

15        The aqueous phase ingredient for producing this aqueous gel core is an active component and other ingredients comprising water and has hydrophilic properties. As the active component, antiseptic agents such as paraoxybenzoate and phenoxy ethanol; humectants such as 1,3-butylene glycol, 20 dipropylene glycol, ethylene glycol, glycerol, and diglycerol; fresheners such as ethanol and menthol; surfactants; and other pharmacological agents such as vitamin C derivatives can be given. As the aqueous phase ingredient, an oil-in-water emulsion in which oil is emulsified and 25 dispersed in water, and a suspension with particles dispersed in water or an oil-in-water emulsion, and the like can also be used.

The water soluble gellant is an agent for gelling water by dissolving or swelling in water. For example, naturally occurring plant high polymers such as agar, guar gum, locust bean gum, quince seed gum, furcellaran, carageenan, sodium 5 alginate, gellan gum, starch, pectin, and konjak; naturally occurring high polymers of animal origin such as gelatin; half-synthesized high polymers such as methyl cellulose, hydroxymethyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, carboxymethylcellulose, and 10 cellulosic derivatives; synthesized high polymers such as polyvinyl alcohol, carboxy vinyl polymer, alkyl modified carboxy vinyl polymer, sodium polyacrylate, sodium polymethacrylate, and polyvinyl pyrrolidone; water swelling clay minerals such as magnesium sodium silicate, 15 water-containing aluminium silicate, montmorillonite, saponite, hecolite, silicon mica tetraflouride, and the like can be given. These may be used individually or in combination of two or more.

As the water-soluble gellant, from the viewpoint of ease 20 in high speed shearing and freeze-shattering, and improvement in storage stability, that form hard gels with water such as agar, gelatin, carageenan, gellan gum, and magnesium sodium silicate are preferable.

In order to ensure that a sufficient amount of aqueous 25 phase ingredient can be acquired, the content of the water-soluble gellant used in the present invention is preferably 0.1-10% of the aqueous gel, varying depending on

the type of water-soluble gellant used.

When the water-soluble gellant of the present invention is a compound requiring a counter-alkali for gelling water, such as carboxy vinyl polymer, alkyl modified carboxy vinyl polymer, and the like, sodium hydroxide, potassium hydroxide, triethanolamine, diethanolamine, and the like may be used. In this instance, depending on the type of water-soluble high polymer used, the ratio of the water-soluble gellant and the alkali is preferably 1:0.001-1:1.

10 In the present invention, as methods for producing a powdered aqueous gel core using an aqueous gel composed of an aqueous phase ingredient and a water soluble gellant, high speed shearing, freeze-shattering, and the like can be given. As the method of high speed shearing, a high speed pin mill, 15 cutter-mixer, and the like can be given. As a method of freeze shattering, freezing of the aqueous gel using a refrigerant such as liquid nitrogen and then crushing the aqueous gel can be given. For the temperature used during freeze-shattering of the aqueous gel, -20°C to -190°C is preferable depending 20 on the hardness and coagulating point of the aqueous gel. The particle size after freeze-shattering is preferably between about 1 µm and 300 µm.

The hydrophobic particles used to coat the powdered particles of the aqueous gel obtained in this manner function 25 to improve anti-aggregation and storage stability by adhering to or being adsorbed onto the surface. As these hydrophobic particles, particles exhibiting hydrophobicity, hydrophilic

particles that are surface treated using a known hydrophobicizing agent, hydrophobic particles further treated using a hydrophobicizing agent to increase the degree of hydrophobicity, and the like can be used. The particle 5 diameter of these hydrophobic particles must be smaller than the particle diameter of the powdered particles of the aqueous gel. A particle diameter 1/10 or less of the particle diameter of the aqueous gel powdered particles is preferable from the viewpoint of coating efficiency of the surface.

10 Among the hydrophobic particles used in the present invention, as examples of the particles having hydrophobicity, polystyrene powder, polyethylene powder, organopolysiloxane elastomer powder, polymethylsilsesquioxane powder, N-acyl-lysine, polyethylene tetrafluoride resin powder, 15 acrylic resin powder, epoxy resin powder, nylon powder, aluminum stearate, zinc laurate, and magnesium stearate can be given. These particles may be used individually or in combination of two or more.

20 A hydrophobic powder prepared by treating the surface of a hydrophilic powder with a hydrophobicizing agent can also be used. As the hydrophobicizing agent, organic silicon compounds such as trimethylsilylation agent and methylhydrodiene polysiloxane, fluorine compounds such as perfluoropolyether alkyl phosphate and perfluoropolyether 25 silane, metallic soaps, oil agents, and the like can be given. These agents may be used individually or in combination of two or more. Of these, organic silicon compounds and fluorine

compounds are preferable due to the improvement in the degree of hydrophobicity. As the hydrophilic particles, for example, inorganic particles such as titanium oxide, zinc oxide, silicic acid anhydride, aluminium oxide, magnesium oxide, zirconium oxide, magnesium carbonate, calcium carbonate, aluminium silicate, magnesium silicate, magnesium aluminium silicate, mica, synthetic mica, synthetic sericite, sericite, talc, silicon carbide, barium sulfate, boron nitride, bismuth oxychloride, and mica titanium; organic particles such as silk powder, starch, and cellulose crystal; and composite particles such as mica titanium coated with titanium oxide powder, zinc oxide powder, or barium sulfate can be given. These hydrophilic particles surface treated with a hydrophobicizing agent may be used individually or in combination of two or more.

Of the above-mentioned hydrophobic particles, fumed silicic acid anhydride treated by a hydrophobicizing agent with an average particle diameter of 0.001-0.1  $\mu\text{m}$  is particularly preferable due to the increase in storage stability. As commercially available products of these hydrophobic particles, AEROSIL R974, R972, RX200, RX300 (manufactured by Nippon Aerosil Co., Ltd.), CAB-O-SIL TS-530 (manufactured by Cabot Corporation), and the like can be given. In the water-containing powder composition of the present invention, a preferable mass ratio of the aqueous gel powder and hydrophobic particles is approximately 100:0.5-100:25, although such a ratio varies according to the particle diameter of the aqueous gel powder and the hydrophobic particles.

There are no particular limitations to the method of coating the surface of the aqueous gel powder with the hydrophobic particles. One example of such a method of coating comprises placing the hydrophobic particles in a stirring 5 vessel and adding the aqueous gel powder while stirring at a low temperature to prevent fusion or aggregation due to a rise in temperature. The stirring vessel used preferably has a jacket with a cooling mechanism and stirring blades that rarely come in contact with the wall and bottom of the jacket.

10 In the cosmetic preparation of the present invention, the content of the above water-containing powder composition is preferably 10-100%, and more preferably 30-90%. When this range is used, a cosmetic preparation exhibiting particularly superior freshness and a refreshing feeling, which are the 15 effects attainable by the addition of water, can be obtained.

10 In addition to the above water-containing powder composition, various additives conventionally used in cosmetic preparations can be added to the cosmetic preparation containing the water-containing powder composition of the 20 present invention to the extent the effect is not adversely affected. Such additives include powders, oil agents; surfactants; oil gelling agents such as partially cross-linked organopolysiloxane and dextrin fatty acid esters; UV absorbers; oil soluble film-forming agents such as 25 acryl-modified silicone and trimethylsiloxy silicate; solvents such as ethanol; antiseptic agents such as para-oxybenzoic acid derivatives and phenoxyethanol;

vitamins; antiphlogistines; antioxidants; chelating agents; pharmacological agents such as vitamin C derivatives; humectants such as glycol; water; water soluble high polymers; polyhydric alcohols; refreshers; and perfumes.

5        Of the powders that can be added to the cosmetic preparation of the present invention, those normally added to cosmetic compositions for the purpose of coloring effect, makeup effect, ultra-violet radiation shielding effect, and feel adjustment effect such as inorganic particles,

10      photoluminescent particles, organic particles, pigment particles, and composite particles, with no limitations to particle shape, size, and structure, can be given. These particles may be used individually or in combination of two or more. Specifically, inorganic particles such as titanium

15      oxide, ferric ferrocyanide, ultramarine, red iron oxide, yellow iron oxide, black iron oxide, zinc oxide, aluminium oxide, silica, magnesium oxide, zirconium oxide, magnesium carbonate, calcium carbonate, chromium oxide, chromium hydroxide, carbon black, aluminium silicate, magnesium

20      silicate, magnesium aluminium silicate, mica, synthetic mica, synthetic sericite, sericite, talc, kaolin, silicon carbide, barium sulfate, bentonite, smectite, boron nitride, and the like; photoluminescent particles such as bismuth oxychloride, mica titanium, iron oxide coated mica, iron oxide, titanium

25      oxide, organic pigment-treated mica titanium, aluminum powder, and the like; organic particles such as nylon powder, polymethyl methacrylate, acrylonitrile-methacrylate

copolymer powder, vinylidene chloride-methacrylic acid copolymer powder, polyethylene powder, polystyrene powder, organopolysiloxane elastomer powder, polymethylsilsesquioxane powder, polytetrafluoroethylene 5 powder, wool powder, silk powder, cellulose crystal, magnesium stearate, zinc stearate, N-acyl-lysine, and the like; pigment particles such as organic tar type pigment, lake pigment, and the like; and composite particles such as mica titanium coated with titanium oxide powder, zinc oxide powder, barium sulfate, 10 titanium oxide containing silica dioxide, zinc oxide containing silica dioxide, and the like can be given. These particles may be used individually or in combination of two or more.

Composite powders made from two or more of the above 15 particles may be used. In addition, particles with the surface treated by a fluorine compound, silicon containing oil agent, metallic soap, wax, surfactant, fat, oil, hydrocarbon, or the like by a conventional method may also be used. The amount of these particles incorporated in the cosmetic preparation 20 of the present invention is preferably 1-90%, although the specific amount varies depending on the purpose of adding the particles and the type of cosmetic preparation.

Oil agents added to conventional cosmetic preparations with the purpose of improving adhesion to the skin, providing 25 emollience, and improving makeup durability may be used in the cosmetic preparation of the present invention. Such oil agents include hydrocarbons, fats and oils, waxes, hardened

oils, ester oils, fatty acids, higher alcohols, silicone oils, fluorine-containing oils, and lanolin derivatives, regardless of the origin (animal oils, vegetable oils, or synthetic oils) and the state (solid, half-solid, liquid, or volatile).

5 Specific examples of such oil agents include hydrocarbons such as paraffin wax, ceresin wax, ozokerite, microcrystalline wax, Japanese tallow, montan wax, fisher tropesch wax, polyethylene wax, liquid paraffin, petroleum jelly, squalane, and the like; natural products such as carnauba wax, beeswax, lanolin wax,

10 candelilla, and the like; esters such as glyceryl tribehenate, pentaerythritol colophonate, pentaerythritol rhodinate, isopropyl myristate, dialkyl carbonate, glyceryl trioctanoate, diglyceryl tri-isostearate, and the like; alkyl modified silicones such as stearyl siloxane and the like; fatty acids

15 such as stearic acid, 12-hydroxy stearic acid, behenic acid, oleic acid, and the like; higher alcohols such as cetanol, stearyl alcohol, behenyl alcohol, and the like; fats and oils such as olive oil, castor oil, jojoba oil, mink oil, and the like; lanolin derivatives such as isopropyl lanolin fatty acid,

20 lanolin alcohol, and the like; silicone oils such as dimethyl polysiloxane, methylphenyl polysiloxane, and the like; cyclic silicones such as decamethyl cyclopentasiloxane, octamethyl cyclotetrasiloxane, and the like; polyoxyalkylene modified and alkyl modified silicone oils; fluorine-containing oils

25 such as perfluorodecane, perfluorooctane, and the like. These oil agents may be used individually or in combination of two or more.

The amount of these oil agents used in the cosmetic preparation of the present invention is preferably 0.1-50%, although the specific amount varies depending on the purpose of addition and the type of cosmetic preparation.

5 The cosmetic preparation incorporating the water-containing powder composition of the present invention includes, but is not limited to, makeup such as eye color, foundation, rouge, face powder, skin care cosmetic preparations such as body powder, anti-perspirant powder, 10 whitening powder, milky lotion, cream, beauty lotion, and sunscreen lotion.

The cosmetic preparation of the present invention explained above containing a water-containing powder composition which breaks and releases water when applied with 15 the fingers, palm of the hand, sponge, or chip applicator possesses the characteristics of refreshing feeling and film forming during use.

The water-containing powder composition of the present invention has excellent production stability and storage 20 stability, breaks and releases water when applied with the fingers, palm of the hand, sponge, or chip applicator. The cosmetic preparation including the water-containing powder composition of the present invention, though having a powdery and solid appearance, easily breaks to release water thereby 25 having refreshing feeling and film forming characteristics that cannot be obtained in conventional cosmetic preparations. Also, in addition to cosmetics, the water-containing powder

composition of the present invention can be used in a broad range of products including foods, perfumes, agricultural chemicals, and medicines.

## 5 EXAMPLES

The present invention will be described in more detail by way of Examples which should not be construed as limiting the present invention.

### 10 Example 1

Water-containing powder composition:

Water-containing powder compositions were prepared in the following manner from the components shown in Table 1. The water-containing powder compositions thus prepared were

15 evaluated for "production stability", "storage stability", and "ease of collapsing and releasing water under finger pressure during use" in the following manner. The results are shown in Table 2.

(Composition)

Table 1

Present Invention  
(weight%)

	Present Invention							Comparative Example	
	1	2	3	4	5	6	7	1	
1. Purified water	Balance	Balance							
2. Agar	0.2	2	2	-	-	-	-	1	-
3. Gelatin	-	-	-	3	-	-	-	-	-
4. Carageenan	-	-	-	-	0.05	0.05	-	-	-
5. Starch	-	-	-	0.2	-	-	-	-	-
6. Carboxy vinyl polymer	-	-	-	-	0.7	-	-	-	-
7. Magnesium sodium silicate *1	-	-	-	-	-	-	-	10	-
8. Alkyl modified carboxy vinyl polymer *2	-	-	-	-	-	0.5	-	-	-
9. Glycerol	2	2	2	2	2	2	2	2	2
10. 1,3-Butylene glycol	10	10	10	10	10	10	10	10	10
11. Antiseptic agent	Appropriate amount	Appropriate amount							
12. Triethanol amine	-	-	-	-	0.7	0.5	-	-	-
13. Liquid paraffin	-	-	-	-	-	5	-	-	-
14. Talc	-	-	-	-	3	-	-	-	-
15. Soybean phospholipid	-	-	-	-	0.3	-	-	-	-
16. Perfume	Appropriate amount	Appropriate amount							
17. Hydrophobicized silicic acid anhydride *3	3	3	25	-	-	-	-	-	-
18. Hydrophobicized silicic acid anhydride *4	-	-	-	3	3	3	3	3	3

\*1: Laponite XLG (manufactured by Laponite Co., Ltd.)

\*2: Carbopol 1342 (manufactured by Goodrich Chemical Co., Ltd.)

\*3: AEROSIL R972 (dimethyl dichlorosilane treated silicic acid anhydride manufactured by Nippon Aerosil Co., Ltd.)

\*4: AEROSIL RX300 (hexamethyl disilazane treated silicic acid anhydride manufactured by Nippon Aerosil Co., Ltd.)

(Method of preparation)

- A. The components 1-12 are mixed and dissolved (or swelled).
- B. The components 13-16 are added to A and are mixed by dispersion.
- C. B is cooled to -80°C using liquefied nitrogen and freeze-shattered to obtain an aqueous gel powder with a particle diameter of 80 µm.
- D. C is added to and mixed with the component 17 or 18 in a stirring vessel while stirring to obtain a water-containing powder composition.

(Method of evaluation)

Production Stability:

The water-containing powder composition was prepared five times under the same conditions and the fluctuation of the particle diameter was evaluated according to the following standard.

Evaluation Standard

- ◎ Completely uniform with no fluctuation
- Nearly uniform with slight fluctuation
- △ Fluctuation is present
- × Fluctuation is significant

Storage stability:

The water-containing powder composition was stored at 40°C for six months and then evaluated according to the following standards.

Evaluation Standard

- Ⓐ No change in outward appearance after storage
- Ⓑ Slight association of the particles is noticeable
- △ Association of the particles is noticeable

5           × Water separation is noticeable

Ease of collapsing and releasing water under finger pressure  
during use:

Each water-containing powder composition was pressed with the fingers, and the condition of water release was

10       evaluated according to the following standards.

Evaluation Standard

- Ⓐ The composition immediately collapses and releases water
- Ⓑ Although slightly difficult to collapse, the composition releases water
- △ Although difficult to collapse, the composition releases water

15           × The composition collapses and releases water with difficulty

20       (Results)

Table 2

	Production stability	Storage stability	Ease of collapsing and releasing water during use
Example 1	○	○	○
Example 2	◎	◎	◎
Example 3	○	◎	○
Example 4	◎	◎	◎
Example 5	○	○	○
Example 6	○	○	○
Example 7	○	○	○
Comparative Example 1	△	×	○

As is clear from Table 2, the water-containing powder compositions 1-7 of the present invention have excellent "production stability", "storage stability", and "ease of collapsing and releasing water under finger pressure during use" in comparison to the Comparative Example.

10 Example 2

Water-containing powder composition:

Water-containing powder compositions were prepared from the following components using the following method. The water-containing powder compositions prepared were evaluated as ◎ for "production stability", "storage stability", and "ease of collapsing and releasing water under finger pressure during use" under the same conditions in Example 1.

(Component)	weight%
1. Purified water	balance
2. Agar	1
3. Magnesium sodium silicate * <sup>1</sup>	5
5 4. Glycerol	2
5. 1,3-butylene glycol	10
6. Antiseptic agent	appropriate amount
7. Triethanol amine	1
10 8. Stearic acid	2
9. Glycol monostearic acid	1
10. Cetanol	1
11. Dimethyl polysiloxane	1
12. Liquid paraffin	5
15 13. 2-ethylhexyl paramethoxy cinnamic acid	1
14. Sorbitan sesquioleate	0.5
15. Polyoxyethylene sorbitan monooleate	0.5
16. Nylon powder	1
20 17. Perfume	appropriate amount
18. hydrophobic treated silicic acid anhydride * <sup>4</sup>	3

(Method of preparation)

25 A. The components 1-7 are mixed (or swelled) in a solution.  
 B. The components 8-15 are heated, dissolved, added to A, and emulsified.

C. The components 16-17 are added and mixed by dispersion.

D. C is cooled to -120°C using liquefied nitrogen, and freeze-shattered to obtain an aqueous gel powder with a particle diameter of 80  $\mu\text{m}$ .

5 E. D is added to and mixed with the component 18 in a stirring vessel while stirring to obtain a water-containing powder composition.

Example 3

10 Deodorant powder:

(Component)	weight%
1. Aluminum hydroxychloride	20
2. Purified water	balance
3. Agar	3
15 4. Propylene glycol	5
5. Polyoxyethylene (20 mol) oleyl ether	0.5
6. Ethyl alcohol	5
7. Hemlock benzal chloride	0.2
8. Perfume	0.3
20 9. Hydrophobic treated silicic acid anhydride * <sup>4</sup>	5

(Method of preparation)

A. 1-4 are mixed and dissolved.

B. 5-8 are mixed and dissolved.

25 C. A is added to B and solubilized.

D. C is powderized using a Henschel mixer (manufactured by Mitsui Miike Chemical Industry Co., Ltd.).

E. D is added to and mixed with 9 to obtain a deodorant powder.

Example 4

5 Wine powder:

	(Component)	weight%
1.	Purified water	10
2.	Agar	1
3.	Wine	80
10	4. Silicic acid anhydride treated with magnesium stearate	3

(Method of preparation)

- A. 1-2 are heated and dissolved, 3 is added and mixed.
- B. A is cooled to -80 to -100°C and then freeze-shattered.
- 15 C. B is added to and mixed with 4 to obtain a wine powder.

Preparation Example 1

Water-containing powder composition (foundation):

	(Component)	weight%
20	1. Titanium oxide treated with fluorine compound * <sup>5</sup>	5
	2. Bengala treated with fluorine compound * <sup>5</sup>	0.1
	3. Yellow iron oxide fluorine compound * <sup>5</sup>	1
25	4. Black iron oxide fluorine compound * <sup>5</sup>	0.05

5.	Sericite treated with silicon * <sup>6</sup>	balance
6.	Talc treated with silicon * <sup>6</sup>	10
7.	Water-containing powder	70
composition of Example 1		

5 \*<sup>5</sup>: powder treated with 5% perfluoroalkyl phosphate

\*<sup>6</sup>: powder treated with 3% methyl hydrodiene polysiloxane

#### Preparation Example 2

Water-containing powder composition (eye shadow)

10	(Component)	weight%
	1. Water-containing powder	balance
	composition of Example 1	
15	2. Partially cross-linked organopolysiloxane swelling material * <sup>8</sup>	2
	3. Dimethyl polysiloxane	1
	4. Silicon treated silicic acid anhydride * <sup>6</sup>	5
20	5. Silicone treated red pigment no. 202 * <sup>6</sup>	0.3
	6. Yellow iron oxide	1
	7. Titanium mica treated with fluorine compound * <sup>5</sup>	5
25	8. Talc treated with metallic soap * <sup>9</sup>	10
	9. Titanium oxide	1
	10. Antiseptic agent	proper amount

\*<sup>8</sup>: silicon KSG-18 (manufactured by Shin-Etsu Chemical

Co., Ltd.)

\*<sup>9</sup>: powder treated with 3% zinc laurate

Preparation Example 3

5 Water-containing powder composition (beauty lotion)

	(Component)	weight%
1.	Water-containing powder	balance
	composition of Example 2	
2.	Cane sugar fatty acid ester	1
10	3. Dipropylene glycol	1
	4. Polystyrene powder	3
15	5. Antiseptic agent	appropriate amount

15 Preparation Example 4

Water-containing powder composition (whitening powder):

	(Component)	weight%
1.	Water-containing powder	balance
	composition of Example 1	
20	2. Ascorbic acid magnesium phosphate	1
	3. Ascorbic acid sodium phosphate	1
	4. Diglycerine	0.5
	5. Hydrogenated soybean phospholipid	1
	6. Squalane	2
25	7. Dextrin fatty acid ester	0.3
	8. Nylon powder treated with fluorine compound * <sup>5</sup>	3

9. Perfume appropriate amount

10. Antiseptic agent appropriate amount

<sup>5</sup>: powder treated with 5% perfluoroalkyl phosphate

### Preparation Example 5

## Water-containing powder composition (sunscreen)

(Component) weight%

10 1. Water-containing powder balance  
composition of Example 2

## 2. Titanium oxide particles treated with fluorine compound <sup>\*5</sup>

### 3. Silicon treated zinc oxide

15 particles  $\times 6$

4. 2-Triglyceryl ethyl hexanoic acid 1  
5. 4-Tert-butyl-4'-methoxy 0.05

## dibenzoylmethane

6. Alkyl polyacrylate powder	5
20 7. Perfume	appropriate

8. Antiseptic agent appropriate amount

\*<sup>5</sup>: powder treated with 5% perfluoroalkyl phosphate

\*<sup>6</sup>: powder treated with 3% methyl hydrodiene polysiloxane

Preparation Example 6

Water-containing powder composition (body powder)

(Component)	weight%
1. Water-containing powder	balance
5 composition of Example 3	
2. 1-Menthol	0.05
3. Camphor	0.05
4. Ethanol	2
5. Silicic anhydride	10
10 6. Antiseptic agent	appropriate amount

## WHAT IS CLAIMED IS:

1. (amended) A water-containing powder composition comprising aqueous gel cores, obtained by gelling an aqueous phase ingredient with a water-soluble gellant, coated with hydrophobic particles, wherein the aqueous gellant is one or more components selected from the group consisting of agar, gelatin, carageenan, gellan gum, and magnesium sodium silicate.

2. (amended) The water-containing powder composition of claim 1, wherein the aqueous gel cores are obtained by gelling an aqueous phase ingredient with a water-soluble gellant, which is one or more components selected from the group consisting of agar, gelatin, carageenan, gellan gum, and magnesium sodium silicate, and freeze-shattering the gel.

3. The water-containing powder composition of claim 1 or claim 2, wherein the hydrophobic particles have a particle diameter of 1/10 or less of the particle diameter of the aqueous gel cores.

4. (Deleted)

5. (amended) The water-containing powder composition of any one of claims 1-3, wherein the cores obtained from the aqueous gel are powdered gel cores.

6. (amended) A process for manufacturing a water-containing powder composition comprising gelling an aqueous phase ingredient with a water-soluble gellant, which is one or more components selected from the group consisting of agar, gelatin, carageenan, gellan gum, and magnesium sodium silicate, to form aqueous gel cores, and coating the aqueous gel cores with hydrophobic particles.

7. The process for manufacturing the water-containing powder composition of claim 6, wherein the aqueous phase ingredient caused to gel using a water-soluble gellant is formed into powdered aqueous gel cores by freeze-shattering.

8. A cosmetic preparation comprising the water-containing powder composition according to any one of claims 1 to 5.

9. A method of applying makeup, comprising applying the cosmetic preparation of claim 8 to the skin and applying pressure to cause water to release from said composition.

ABSTRACT OF THE DISCLOSURE

A water-containing powder composition comprising particles each composed of an aqueous gel as a core and 5 hydrophobic particles with which the core is coated; and a process for producing the water-containing powder composition which comprises causing an aqueous phase ingredient to gel with a water-soluble gellant, forming the gel into particles each serving as a core, and then coating each core with hydrophobic 10 particles.

The composition is characterized in that it is excellent in production stability and storage stability and that the gel-based particles break to release water when used and pressed with, e.g., fingers. The water-containing powder 15 composition is widely utilizable in cosmetics, foods, perfumes, agricultural chemicals, medicines, etc.

# Declaration and Power of Attorney For Patent Application

## 特許出願宣言書及び委任状

### Japanese Language Declaration

#### 日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者（下記の名称が複数の場合）であると信じています。

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As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

WATER-CONTAINING POWDER COMPOSITION, PROCESS  
FOR PRODUCING THE SAME, AND COSMETIC PREPARATION  
CONTAINING THE POWDER COMPOSITION

the specification of which

is attached hereto.  
 was filed on August 17, 2000  
as United States Application Number or  
PCT International Application Number  
PCT/JP00/05510 and was amended on  
July 16, 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

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Prior Foreign Application(s)

外国での先行出願

237149/1999 (Number) (番号)	Japan (Country) (国名)
172172/2000 (Number) (番号)	Japan (Country) (国名)

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I hereby claim foreign priority under Title 35, United States Code, Section 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Claimed 優先権主張	
<input checked="" type="checkbox"/> Yes はい	<input type="checkbox"/> No いいえ
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(Status: Patented, Pending, Abandoned) (現況:特許許可済、係属中、放棄済)
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

## Japanese Language Declaration (日本語宣言書)

委任状：私は下記の発明者として、本出願に関する一切の手続きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。  
(弁護士、または代理人の指名及び登録番号を明記のこと)

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## Japanese Language Declaration

(日本語宣言書)

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